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SRL RECORD COPY PUREX: PROCESS AND EQUIPMENT PERFORMANCE\*

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#### INTRODUCTION

The Purex process is the solvent extraction system that uses tributyl phosphate (TBP) as the extractant for separating uranium and plutonium from irradiated reactor fuels. Since the first flowsheet was proposed at Oak Ridge National Laboratory in 1950, the process has endured for over 30 years with only minor modifications. The spread of the technology was rapid, and worldwide use or research on Purex-type processes was reported by the time of the 1955 Geneva Conference. The overall performance of the process has been so good that there are no serious contenders for replacing it soon.

Purex has also had a substantial affect on solvent extraction science. The desire by many establishments and countries to investigate nuclear materials processing has led to a large investment in fundamental research. This research has covered the mechanisms of extraction, metal-organic compounds, mixing phenomena, thermodynamics, kinetic effects, interphase transfer, analytical techniques, and many other areas of direct application to solvent extraction.

The many papers of the McKay group at Harwell are examples of the contributions. This is not to say that Purex is responsible for the major advances in solvent extraction technology, just that the association with nuclear processing has made funding easier for solvent extraction research.

Two papers on nuclear fuel processing, given at these Conferences in the past, would be difficult to improve on. Spence, at ISEC '71, covered the early history of solvent extraction for nuclear materials recovery. A subsequent comprehensive description of some of the varied approaches was given by A. Chesne' at ISEC '80. I will discuss the evolution of the Purex system, the comparative performance over the years, the areas of science and engineering that have been involved, and some possible areas of continued development.

Some other Purex-related systems that utilize different strengths of TBP for the separation of heavy elements can be operated in the equipment used for Purex. Indeed, one of the original Savannah River Plant (SRP) Purex plants has operated with the following variations:

- Thorex, which separates and recovers thorium target material and U-233
- Interim 23, which recovers only U-233, and sends thorium to waste
- 25 Process, which recovers irradiated highly or medium enriched uranium
- Assorted flowsheets for recovery of higher isotopes of plutonium from irradiated plutonium fuel
- Assorted flowsheets for recovery of transplutonium elements.

<sup>\*</sup> The information contained in this article was developed during the course of work

The ranges of TBP strength utilized in these processes have ranged from 1.5% to 42% at different sites.

#### PUREX PROCESS DESCRPITION

A brief outline of the basic Purex process is warranted. The first cycle involves coextraction and decontamination of uranium and plutonium in a first contactor, reductive partition of plutonium from uranium in a second contractor, and recovery of the uranium in a third contactor. Separate second cycles then give further decontamination of the plutonium and uranium. The spent solvents are washed with alkaline solutions and recycled. Many variations of this basic outline have been utilized on a large variety of irradiated fuels. Extraction contactors are generally center-fed units with an extraction section and a scrubbing section for decontamination, while stripping contactors are generally end-fed. Center-fed contactors can be and have been replaced by two end-fed units. Extra cycles have been provided in some installations to obtain higher decontamination with less restrictive conditions. Other flowsheet variations include backcycle and reflux flowsheets. In backcycle, overall waste volumes can be reduced by utilizing the waste from one cycle as chemical adjustment for a previous cycle. Solvent streams can also be backcycled to reduce losses or add some operating versatility.

Reflux flowsheets have been used to obtain high concentrations by recycling a product stream back to a feed stream and drawing off a small product stream after the desired concentration has been reached. Finally, completely separate solvent systems may be provided for each cycle, or two or more may be combined. The net result of all these possibilities is that few, if any, Purex installations have had exactly the same flowsheets or cycle arrangements. However, the many variations have all had at least satisfactory performance.

### **EQUIPMENT PERFORMANCE**

The requirements for a high decontamination factor (at least 10'), low losses of valuable fissile materials, and minimum space for very expensive shielded facilities, mean that the solvent extraction contactors must be highly efficient and reasonably compact. The contactors primarily utilized for Purex operations have been pulse columns and mixer-settlers, with some limited use of centrifugal units. Pulse columns were ushered in with Purex processing in 1949 and 1950 at Oak Ridge National Laboratory, following development work at Hanford and Oak Ridge in the recovery of uranium from stored waste. The emergence of pulse columns at this time is illustrated by the 1950 edition of Perry's Chemical Engineers' Handbook, which does not even include pulse columns in its discussion of contactor types, even though a patent for the concept was issued in 1935.

Mixer-settlers, on the other hand, have been used in a variety of sizes and configurations following the first description of one in 1904 (which was amazingly similar to some present day units). Overall, mixer-settlers in production work have ranged from small units for enriched uranium and plutonium-rich fuels (as at Eurochemic and Dounreay), to large units like those at Sellafield and Savannah River. Even these units are very small compared to industrial units.

Pulse columns and assorted designs of mixer-settlers were adopted as the number of Purex installations grew. The Savannah River Plant adopted mixer-settlers of a pump-mix design (developed at Knolls Atomic Power Laboratory) for its two Purex plants that began operating in 1954 and 1955. The Hanford Purex Plant, which began operating in 1956, utilizes scaled-up pulse columns. Many other installations that use pulse columns and mixer-settlers were described in the Geneva Conferences of 1955, 1958, and 1964, and at ISEC conferences.

### Mixer-Settlers

General factors that determine the efficiency of mixer-settlers have been reviewed many times. Some of the earliest production Purex units were those at Savannah River. The KAPL pump-mix impeller design moves the heavy phase through the bank, and balances the mixing and pumping functions to give good efficiency at a restricted flowrange. Above the design range, the high impeller speeds necessary

for high flows result in excessive mixing, with consequent excessive entrainment in the reduced residence time that accompanies high flow. Below the design range, low impeller speeds for low flow provide insufficient mixing and low efficiency. Units with separate pumping and mixing functions in a variety of configurations are used at other installations. Such units can maintain a high stage efficiency and reasonable entrainment over a wide flow range.

Mixer-settler stage efficiencies in most Purex applications can be held at 70% to near 100% relatively easily where the bulk transfer of uranium and plutonium is involved. Efficiencies can be low in extreme flow ratio regions, such as some scrub sections, where transfer of a small amount of material from the bulk phase into a small volume of dispersed phase is desired.

One requirement for the continued efficient operation of mixer-settlers is that solids not accumulate in the banks, particularly at the interfaces in the settlers. Such material can cause excessive solvent degradation if fission products accumulate and become a localized high radiation source. Such a phenomenon involving fission product zirconium was reported from Marcoule; it was solved by the addition of fluoride ion, which dissolved the zirconium. Such behavior appears to depend on the characteristics of the feed solutions and specific flowsheets. Mobile solids in the aqueous phase can move through some banks without accumulations. Also, no large solids problem has been found at Savannah River, where all feed is centrifuged for clarification after treatment to coagulate silicic acid, which originates from aluminum associated with the fuels.

#### Pulse Columnms Performance

Efforts to increase the efficiency of pulse columns have continued since their inception in Purex service. Many variables have been explored: amplitude, frequency, and shape of the pulses; size, number, and shape of sieve-plate holes; configuration and spacing of plates; wetting characteristics of surfaces, etc. For the nuclear applications specifically, a high efficiency is necessary to avoid undue height that must be enclosed in heavily shielded facilities. To decrease overall height, columns that might be center-fed in a small-scale installation have been divided into two end-fed units. The height of an equivalent theoretical stage increases somewhat with column diameter. However, actual individual columns that have been built are in the same height range for plants of considerably different capacity. Eurochemic, the Barnwell plant (at five-tons-per-day design basis), and the Hanford plant all had assorted columns in the 10- to 15-meter range, with diameters for the largest in each plant ranging from 150 mm to 812mm.

Pulse columns have many favorable features for nuclear fuel processing. First-stage extraction columns tolerate substantial solids in the feed when operated organic-continuous with a bottom organic-aqueous interface. The solids follow the aqueous down and out of the column, and the organic overflow is quite clean. Such operation has become essentially standard. Also, columns with reasonable throughputs have been designed and operated with diameters favorable for nuclear criticality safety, where concentrated fissile materials have been present. Additionally, relatively short residence times limit the amount of solvent degradation when highly radioactive solutions are being processed. Overall, pulse columns have provided good service in many Purex installations.

### Rapid Contactors

Rapid contactors that utilize centrifugal force to aid in separating the phases allow rapid startup and shutdown. In Purex operations, the two primary types of contactors whose uses have been reported are the SGN-Robatel multi-stage unit, and the Savannah River single-stage unit which is arranged in banks. The Robatel unit can provide high efficiency, but demands highly clarified feed since solids can clog the small passages. Test work in Purex applications at Marcoule and other CEA facilities was reported at ISEC '71. Good efficiency and uranium capacities of 5 and 8 tons per day were reported for different units. A Robatel unit was installed as the first contactor in the Allied General Nuclear Services Barnwell Plant, but no performance data in this demanding service have been obtained because the plant did not start up.

An 18-stage bank of the Savannah River centrifugal contactors was installed in 1966 as a replacement for the large mixer-settler bank that had demonstrated large solvent degradation effects. As reported in 1971, the unit provided low solvent degradation and allowed rapid startup and shutdown. The small holdup volume and rapid response have made the standard discontinuous operation, with weekly startup and shutdown, relatively simple. The contactor has given good mechanical service. The primary reason for replacing a motor unit has been excessive leakage of the weir-control air at the rotating seal, although many seals have lasted ten years or more (one operated for 18 years). Still, new bearings and seals that should extend life have been designed and are being installed. Vibration analysis is also recommended. A fatigue failure at a weld occurred at the same position on each of the six-stage sub-units after 7, 10, and 13 years respectively. Reinforcement appears to have solved the problem.

Kinetic effects still appear to limit zirconium decontamination in the nine rapid scrub stages in the bank, as noted in the report at ISEC '71. Only recently has further experimental work started to explore the effect of slow scrubbing stages following the rapid centrifugal stages.

A variation of the single-stage centrifugal contactor has been introduced by Argonne National Laboratory personnel and developed further at Savannah River. Mixing takes place in the annular space between the bowl and housing, and the seal for the air for interface control has been simplified. Banks with bowls in a range of sizes have been built, but none have been used in production operations yet.

### FISSION PRODUCT DECONTAMINATION

The fission product elements that determine the final decontamination that can be attained in the Purex process are ruthenium, zirconium, and niobium. As a result, many studies have been undertaken to understand the fundamental chemistry and extraction behavior of these elements. Ruthenium has been found to form a large number of nitroso and nitro species in aqueous nitrate solutions. These species have different distribution coefficients and relatively slow equilibration times between species. Further, slowly equilibrating complexes with TBP are formed in the organic phase. The additional effects of degraded solvent on ruthenium decontamination are generally small, but they have been important in a few cases. The zirconium problems have been due primarily to interactions with solvent degradation products. The simplest of these is the well-known complex with dibutyl phosphate. However, the limits on performance with highly active feed result from interactions with the other components of degraded solvent. Studies on the fission products continue, as attested to by papers at this conference. But note again, that lack of knowledge has not prevented 35 years of successful operation.

Differences in fission product performance have been reported with different flowsheets and equipment. At Savannah River, the original, small 16-stage mixer-settler banks provided about the same first-cycle decontamination as the Oak Ridge Pilot Plant pulse columns. The small 1A 16-stage bank in one plant was replaced with a 24-stage, jumbo bank as part of a general plant revision in 1959. As reported at the 1962 Gatlinburg Symposium, the many long-residence stages initially gave excellent decontamination factors for zirconium and ruthenium (up to 2 x 10<sup>4</sup> for zirconium and near 10<sup>5</sup> for ruthenium). However, the 7- to 15-minute-per-stage residence times caused serious solvent degradation problems that resulted in low decontamination and high solvent activity. Improved solvent washing, dodecane diluent, and limitations on feed activity finally provided a stable, but low, decontamination. In 1966, an 18-stage bank of centrifugal contactors replaced the jumbo 1A bank. This solved the solvent degradation problem and improved decontamination somewhat, but the fewer, short-residence scrub stages limit decontamination.

Zirconium presented a different problem at Marcoule, as reported at ISEC '71. Zirconium-accumulating solids gathered in the mixer-settlers, accelerating solvent degradation and decreasing the decontamination. A small concentration of fluoride was added to the feed to complex the zirconium. The fluoride eliminated the solids and increased the decontamination factor to greater than 10<sup>4</sup> for zirconium, and to greater than 3000 for ruthenium.

Essentially, all sites have operated at a high degree of saturation of the organic phase by uranium during the initial extraction. This condition leaves less TBP available to complex the fission products and increases the decontamination from fission products markedly. The Windscale Purex Plant represented another departure with a low-saturation flowsheet that accepted a lower fission product decontamination to keep solids and recoverable plutonium out of the waste. At lower saturation, both Pu(IV) and the somewhat less extractable Pu(VI) that may be formed during fuel dissolution are recovered efficiently, resulting in low losses without the addition of valence adjustment chemicals.

Still other systems have provided variations in the initial extraction acidity, scrub acidity, and temperature to improve fission product decontamination. The distribution coefficient of zirconium increases as acid increases, while that of ruthenium decreases as acid increases; hence, compound scrub sections with high and low acid sections are used in some cases to improve decontamination. Also, ruthenium equilibrates more rapidly at high temperatures, allowing it to be scrubbed more efficiently.

### SOLVENT EFFECTS

The nature of the diluent used with the TBP and the treatment of the spent solvent from the process steps are very important to the performance, when decontamination factors on the order of  $10^7$  are needed. Originally, the chief worry was the decomposition of TBP to dibutyl phosphoric acid and monobutyl phosphoric acid by radiolysis and acid hydrolysis. Washes with sodium hydroxide or sodium carbonate solutions to remove these acidic species were part of the original flowsheets. It was found early that dibutyl phosphate complexes zirconium, carrying it through the process, and decreasing the decontamination. The dibutyl phosphate also complexes plutonium strongly and can cause high plutonium losses to the solvent washers.

Soon after production-scale operations began, it became apparent that degradation of the diluent was having a large influence on performance. Other compounds in the solvent besides the butyl phosphates were transporting zirconium, niobium, and ruthenium through the process, reducing decontamination, and causing high levels of activity in the circulating solvent. Several areas of study followed: the relation between the structure of the hydrocarbon diluent and its stability; identification of the degradation products as an aid to their removal; solvent regeneration techniques and alternative washing agents.

Empirical tests in the late 1950's demonstrated that excessive branched chains and aromatic compounds in the hydrocarbons used as diluent could be the precursors of the unidentified degradation products. Straight-chain paraffins in the twelve-carbon range had a desirable combination of density, viscosity, flash point, and radiation resistance. Dodecane became almost a standard to measure other diluents against. Solvent degradation problems led Savannah River to adopt n-dodecane as a diluent in 1961. The first inventories of n-dodecane were quite expensive. Thanks to the demand for biodegradable detergents, supplies of mixed n-paraffins that were predominately dodecane became available at reasonable prices, and their use spread. Note that other hydrocarbon diluents offer advantages where radiation degradation is not a problem. Some metal-TBP complexes, such as plutonium and thorium, have a limited solubility in the hydrocarbon diluent, and at high concentrations will separate out as a heavy organic phase. The complexes are considerably more soluble in aromatic diluents; hence, aromatics may be the diluent of choice in some concentrated flowsheets. Overall, diluent can have substantial influence on the performance of a given system, and process requirements should be considered carefully in its selection.

### Solvent Degradation Products

The identification of the degradation products responsible for the decrease in decontamination has been the subject of investigation and speculation for 25 years. Even as early as the 1962 Gatlinburg Solvent Extraction Chemistry Symposium, one paper had 58 references on solvent degradation. Some of the most definitive work has been done at Karlsruhe, Germany with identification of several classes of compounds formed from the primary activation of the diluent and TBP. These include

carbonyl, nitro, and nitroso compounds from diluent alone, long-chain acid phosphates from diluent and TBP combinations, and polymeric butyl phosphates from TBP interactions. Some of these compounds have the zirconium retention properties observed in plant operations. The conductivity techniques developed at Karlsruhe for determining the amounts of these compounds can be very useful in determining whether poor performance is due to their presence or to some other operating variable.

Sodium carbonate still appears to be the primary solvent cleanup solution in use at nearly all sites reporting. Various other cleaning systems have been tried, such as permanganate added to the alkaline washers and absorption columns of many materials, including ion exchange resins, calcium oxide, lead oxide, and alumina. Given the assortment of compounds that have been found in the solvent, some of them could be removed by almost any of the reagents. Recent Karlsruhe work has utilized hydrazine carbonate as a washing agent that does not contribute to waste solids; a trial of hydrazine carbonate also has been reported at La Hague. The prime advantage of this reagent is the small amount of waste solids generated as compared with sodium carbonate and sodium hydroxide. The solids are important when the cost of final waste disposal is considered.

### PARTITIONING OF URANIUM AND PLUTONIUM

The agents used to reduce Pu(IV) to Pu(III) and partition it from uranium varies between sites. Ferrous sulfamate was the agent in the original Purex flowsheet and is still used extensively. The efforts to reduce waste solids early led to the consideration of U(IV) as a substitute that would be more efficient because it acts on plutonium in both the aqueous and organic phases.

Two methods have been utilized for the reductions with U(IV): the introduction of an externally generated U(IV) solution at multiple positions along the 1B contactor, and the electrolytic generation of U(IV) in situ. Typical utilization of U(IV) solution was reported from Marcoule and Eurochemic at ISEC '71. An early installation for electrolytic reduction was at the WAK mixer-settlers at Karlsruhe for the stripping of plutonium in the second plutonium cycle. Successful test work with both mixer-settler and pulse column units for the first-cycle uranium-plutonium partition also has been reported there. A large pulse column unit was built for the five-ton-per-day Barnwell Plant, but its performance was never determined.

A typical problem with the partitioning of plutonium with either ferrous sulfamate or U(IV) is the large consumption beyond the stoichiometric requirement, leading to significant excess waste with ferrous sulfamate. Considerable study has taken place on the mechanisms of consumption and on methods to reduce the amount of reductant required. Low acid in the bank is favorable, and both hydroxylamine nitrate and hydrazine nitrate can substitute for some ferrous sulfamate.

The overall selection of the method of partitioning rests upon the importance of waste, equipment flexibility in a given installation, the tolerable uranium contamination in the plutonium product, and the uranium separation capability of any subsequent plutonium cycles.

### CONCLUSION

Many different arrangements of the basic Purex process have been designed, involving different equipment and chemicals, with specific details resting on the goals and restrictions set for a given installation. Flowsheets can be designed for almost any required level of product purity, for concentrated or dilute products and for low solids in the waste. Systems are also versatile, capable of processing many different feeds and products. Other processes for reactor fuel processing have been explored, but the Purex process has had no major competitors in the past 35 years, and none are apparent in the foreseeable future.

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February 21, 1986

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A paper proposed for presentation at the International Solvent Extraction Conference-ISEC '86, in Munich, Germany, on September 11-16, 1986.

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Yours sincerely,

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Deutsche Gesellschaft für chemisches Apparatewesen

Prof. D. Behrens

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February 21, 1986

TO:

J. F. SABINO

FROM:

C. J. BANICK

DOCUMENT REVIEW

Document(s) DP-MS-86-28

Title(s): PUREX: PROCESS AND EQUIPMENT PERFORMANCE

Author(s): D. A. Orth

Contractual Origin: DE-AC09-765R00001

Present Classification: Unclassified Paper

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No items were noted that, in my opinion, should be called to the attention the DOE for patent consideration.



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March 13, 1986

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Dear Sir or Madam:

Enclosed please find the camera-ready copy for the paper "PUREX: Process and Equipment Performance" by D. A. Orth. The paper is proposed for presentation at the ISEC '86 Meeting, September 11-16, 1986 at Munchen, West Germany, and for publication in the proceedings.

Also enclosed is a copyright transfer agreement, and two photostatic copies of the manuscript.

If we can be of further assistance please let us know.

Sincerely,

Jeffrey S. Hammond Publications Division

JSH: tww

Enclosure

Title: PUREX: PROCESS AND EQUIPMENT PERFORMANCE

Author: D. A. Orth Editor: Hammond

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